

EFFECT OF CALCINATION TEMPERATURE ON KAOLINITE FOR MULLITISATION OF KAOLINITE AND ALUMINA MIXTURE

A THESIS SUBMITTED BY

Abinash Lenka

In Partial Fulfillment of the Requirement

For the degree of

BACHELOR OF TECHNOLOGY



DEPARTMENT OF CERAMIC ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

ROURKELA, ORISSA - 769008

MAY 2012

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(Under the guidance of Prof. Sunipa Bhattacharya)

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ROURKELA

CERTIFICATE

This is to certify that the project entitled “EFFECT OF CALCINATION TEMPERATURE ON KAOLINITE FOR MULLITISATION OF KAOLINITE AND ALUMINA MIXTURE” submitted by Mr. Abinash Lenka is an authentic work carried out by him under my supervision and guidance. Certified further, to the best of my knowledge, the work contained in this project doesn't form any part of any other thesis or dissertation or has not been submitted to any other university for the award of any degree.

Prof. Sunipa Bhattacharya

Date 11.05.2012

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ABINASH LENKA

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LIST OF FIGURES:

FIGURE NUMBER	FIGURE CAPTION	PAGE NO.
4.1	TG/DSC ANALYSIS OF KAOLINITE	24
4.2	XRD PLOT OF CALCINED KAOLINITE (6000 ⁰ C)	25
4.3	XRD PLOT OF CALCINED KAOLINITE (1400 ⁰ C)	26
4.4	XRD PLOT OF CALCINED ALUMINA	26
4.5	VARIATION OF AP	27
4.6	VARIATION OF BD	28
4.7	VARIATION OF FLEXURAL STRENGTH	29
4.8	XRD OF S1*	30
4.9	XRD OF S2*	31
4.10	XRD OF S3*	32
4.11	XRD OF S4*	32
4.12	COMPARISON OF S1,S2,S3 & S4	33

*S1- Raw kaolinite + calcined alumina

*S4- Calcined kaolinite (600⁰C) + calcined alumina

*S2- Calcined kaolinite (1000⁰C) + calcined alumina

*S3- Calcined kaolinite (1400⁰C) +calcined alumina

LIST OF TABLES:

TABLE NUMBER	TABLE CAPTION	PAGE NO.
3.1	PARAMETERS OF CHEMICAL ANALYSIS OF RAW KAOLINITE	18
3.2	RESULTS OF CHEMICAL ANALYSIS	19
4.1	PHYSICAL AND MECHANICAL PROPERTIES OF SAMPLES	29
4.2	AMOUNT OF MULLITE PRESENT IN THE PRODUCTS	34

CONTENTS

PAGE NO.

Certificate	03
Acknowledgement	04
List of figures	05
List of tables	06
Abstract	09
CHAPTER 1: INTRODUCTION	10
CHAPTER 2: LITERATURE REVIEW	13
CHAPTER 3: EXPERIMENTAL WORK	17
3.1 Raw material characterization	18
3.1.1 Chemical analysis of kaolinite	18
3.1.2 Batch calculation	19
3.1.3 TG/DSC analysis of raw kaolinite	20
3.1.4 Calcination of kaolinite	20
3.1.5 XRD analysis of raw materials	20

3.2 Batch preparation	21
CHAPTER 4: RESULT AND DISCUSSION	23
4.1 TG/DSC of raw materials	24
4.2 XRD analysis of raw materials	25
4.3 Physical and mechanical properties	27
4.4 XRD analysis of final products	30
4.5 Amount of mullite phase in different batches	34
CHAPTER 5: CONCLUSION	35
REFERENCES	37

ABSTRACT:

The present project work reports whether there is any effect of calcination temperature of kaolinite, on the mullitisation behavior of kaolinite and alumina mixture. Initially raw material characterization such as chemical analysis, DSC/TG analysis and XRD were carried out. Further in this experiment kaolinite was calcined at various temperatures (600⁰C, 1000⁰C, 1400⁰C) and calcined alumina powders were added in it, specimens were prepared by pressing and finally all the batches were fired at 1600⁰C. The effect of calcination temperature was studied by performing different experiments, mainly physical properties such as apparent porosity & bulk density; mechanical properties and phase analysis by X-ray diffraction. Finally from the results it has been observed that there is always a significant impact of calcination temperature on the mullitisation behavior of kaolinite and alumina mixture. It has been found that the kaolinite that was calcined at higher temperature show some superior properties when mixed with alumina and fired, as compared to other batches. It shows higher amount of mullite content when fired along with other batches at 1600⁰C, and also better mechanical and sintering properties. The batch that was made by mixing calcined alumina along with raw/uncalcined kaolinite show poor mechanical and sintering behavior.

CHAPTER 1

INTRODUCTION

In past few years mullite has been considered as a very promising material from both refractory material and advanced ceramic material point of view, due to its certain properties such as low density, low conductivity, low expansion coefficient, low dielectric constant, high creep resistance and also excellent mechanical properties at high temperature. There have been many methods in which high pure mullite can be synthesized; it depends upon the raw materials chosen and synthesis route. It can be prepared from sintering of alumina and silica powders by sol-gel method, hydrothermal synthesis, co-precipitation method, chemical vapor deposition etc. but due to costly raw materials and synthesis technique, mullite can't be economically produced in a large scale by these techniques. Due to above reasons kaolinite/clay is still preferred as a starting raw material for mullite formation in addition with alumina. Kaolinite is mainly used because of its common occurrence and cheap availability. When alumino-silicate materials are used, they form mullite above 1400⁰C.

When kaolinite and alumina mixture is heated, it starts forming meta-kaolin (dehydrated kaolin, a meta stable phase) in the range of 500⁰C-600⁰C. It is generally shown in DSC plot by an endothermic peak. At around 980⁰C, a conversion of meta-kaolin into a spinel phase is completed and it is shown by an exothermic peak in the DSC curve. The kaolinite–meta kaolin transformation is a very slowly process and meta kaolin has an extreme defect structure; about 20 vol % of the meta kaolin consists of lattice vacancies produced by the temperature-induced water release [1]. It may be considered as the reason for lower physical and mechanical properties of unclacined kaolinite and alumina mixtures as reported in this project work. At around 1250⁰C-1300⁰C primary mullitisation takes place in the system, which is an exothermic reaction and the added alumina mainly remains inert at least up to 1400⁰C. Beyond that the added alumina reacts with the viscous silica present in the system to produce secondary mullite

[2]. The rate of secondary mullite formation is very low up to 1555⁰C but above 1595⁰C, the growth rate increases rapidly. According to Liu et al. [3], the rapid kinetics of secondary mullite formation, the characteristics of the coexisting glassy phase and the evidence of liquid phase sintering strongly suggest that solution precipitation via a transitory liquid phase was the major mechanism for secondary mullite formation. Secondary mullite formation via solid state interdiffusion may occur in parallel, but its contribution to secondary mullite formation can only be regarded as minor. So, if firing temperature is above 1595⁰C, then the amount of liquid phase increases in the system along with a decrease in the viscosity of the liquid, which indirectly promotes the solution precipitation mechanism.

CHAPTER 2

LITERATURE REVIEW

Mullitisation behavior of three different south Indian clays and three different alumina sources to produce stoichiometric mullite was studied by V.Viswabaskaran et.al. [4] They have found that when the source of alumina is boehmite, then it shows poor mechanical and physical properties, though the aspect ratio of mullite crystal is high. They also have found that the impurities in the raw material improve the strength but not the density of mullite. It was also observed that when the source of alumina is gibbsite then there was a presence of higher amount of secondary mullite crystals than the primary crystals due to presence of impurities. The low strength in case of boehmite arises due to high amount of water release resulting in surface cracks.

Kuo-chang-liu.et.al [5] have studied the effect of temperature on the primary and secondary mullite formation in high purity kaolinite- α alumina mixture by conventional processing. Depending upon temperature primary crystallization, secondary crystallization, crystal growth, formation of co-existing glassy phase etc. occur. They also have found that, at higher temperature ($>1400^{\circ}\text{C}$), evidence of liquid phase sintering strongly support that solution precipitation via transitory liquid phase was the major mechanism for secondary mullite formation, though secondary mullite formation via inter diffusion is also a minor route in the kaolinite α -alumina reaction

M.A Sainz et.al. [6] have carried out a microstructural study of mullite obtained by the reaction sintering of kaolinite and α -alumina mixture in the range of 1150°C to 1700°C . By X-ray profile analysis they have found that between 1350 and 1500°C there is a presence of two different mullite morphologies simultaneously. In that range both maximal frequencies of crystallite size are shifted to greater values, reflecting crystal growth. From XRD microstructural studies they found that bimodal mullite crystal corresponding to secondary mullite nucleation

and primary mullite growth were clearly observed at 1300⁰C. By SEM analysis they found that above 1550⁰C both primary and secondary mullite growth occurs simultaneously and substantial growth is observed above 1700⁰C.

C.Y Chen et.al. [7] have prepared mullite by reaction sintering of kaolinite and alumina. They have studied the phase and microstructural evolution of the specimens and their mechanical properties. They have found that primary mullite appears at around 1200⁰C. The alumina particles are inert up to that temperature and above 1300⁰C they start to react with silica glassy phase to form secondary mullite. Also they have found that, the formation of secondary mullite decreases with amount of glassy phase and furthermore the addition of alumina reduces the size of mullite grains and their aspect ratio. The strength and toughness of resulting mullite increase with increasing alumina content.

V. Viswabaskaran et.al. [2] have studied the mullitisation behavior of calcined clay and alumina mixture. They have performed the experiments using three different varieties of clay along with three different sources of alumina. They have found that, the calcined clay derived samples show better strength and density than the uncalcined clay derived sample. The microstructures also show a higher aspect ratio of mullite crystals.

Joseph A.Pask [8] has studied the stable and meta-stable phase equilibria and reactions in the SiO₂- α Al₂O₃ system. He has found that procedures in the preparation of specimens in this system are critical due to the difficulty of nucleating α -alumina when the silicate liquid is not saturated with alumina even though it may be super saturated with crystalline α -alumina and the ease with which mullite growing from an alumino-silicate melt during cooling accommodates an excess of alumina.

Akshoy K. Chakraborty [9] has mullite formation in a kaolinitic clay occurs by the exhibition of two high temperature exotherms. He has found that there are two simultaneous paths of mullitisation reaction instead of a single solid-state reaction process.

CHAPTER 3

EXPERIMENTAL WORK

The basic objective of this project is to study the effect of calcination temperature of kaolinite on the mullitisation behavior of kaolinite and alumina mixture. If only kaolinite is used as a source of mullite, then after conversion of all the alumina present in the system to mullite some residual silica are left in the system as a glassy phase. So, in order to reduce that glassy phase and to increase the amount of mullite in the system some alumina is added with it.

3.1 RAW MATERIAL CHARACTERISATION

3.1.1 CHEMICAL ANALYSIS OF KAOLINITE

For the chemical analysis of kaolinite sample was taken in a platinum crucible. The parameters for the chemical analysis are given below in table 3.1.

Weight of Na ₂ CO ₃	10gms
No. of NaOH beats	4-5
Rate of heating	3 ⁰ C/min
Fusion temperature	1000 ⁰ C/1hr
Fusing time	1hr
Weight of sample taken	0.4922gm

(TABLE-3.1)

The result of the chemical analysis has been summarized below in the table-3.2.

SiO ₂	52.2 %
Fe ₂ O ₃	0.58 %
Al ₂ O ₃	33.01 %
CaO	1.88 %
MgO	Negligible
LOI	12.4 %

(TABLE-3.2)

3.1.2 BATCH CALCULATION

From the stoichiometric formula of mullite (3Al₂O₃.2SiO₂) it can be seen that mullite consists of 71.79% alumina and 28.21% silica. So, from the calculation we can see that after primary mullitisation the amount of free silica left in the calcined clay system (in 100gm raw sample) will be 44.789 gm. So, extra alumina that will be required to convert all the excess silica into mullite i.e. secondary mullitisation is 113.980gm. So, for 87.6gm of calcined clay 113.980gm of alumina will be required for complete mullitisation.

3.1.3 TG/DSC OF RAW KAOLINITE

TG and DSC analysis of raw kaolinite was carried out in order to know about the temperatures at which the transitions occur so that during firing the schedule can be adjusted.

This was carried out up to 1100⁰C at a rate of 10⁰C/min.

3.1.4 CALCINATION OF KAOLINITE

In order to study the effect of calcination on mullitisation, the raw kaolinite was calcined at different temperatures such as 600⁰C, 1000⁰C and 1400⁰C. The major reason for choosing these temperatures is that after calcining, different types of phases are present in these batches. In the 1st batch meta-kaolin starts forming, so except silica and some alumino-silicate materials nothing can't be expected. In the 2nd batch due to relatively higher calcination temp, in that range a spinel phase is expected which is formed from the meta-kaolin. In the 3rd batch which was calcined at 1400⁰C, mullite phase is expected. So all these three types of phases may have some kind of impact on final mullitisation when it is mixed with alumina and fired, and the objective of the project is to study that with respect to sintering, mechanical properties and phase point of view.

3.1.5 XRD OF RAW MATERIALS

XRD analysis of raw materials used in the above project were carried out, in order to find out the phases present in them and the effect of calcination temperature on mullite.

3.2 BATCH PREPARATION

For a comparative study we have to prepare four different batches with perfect stoichiometric batch calculation such as,

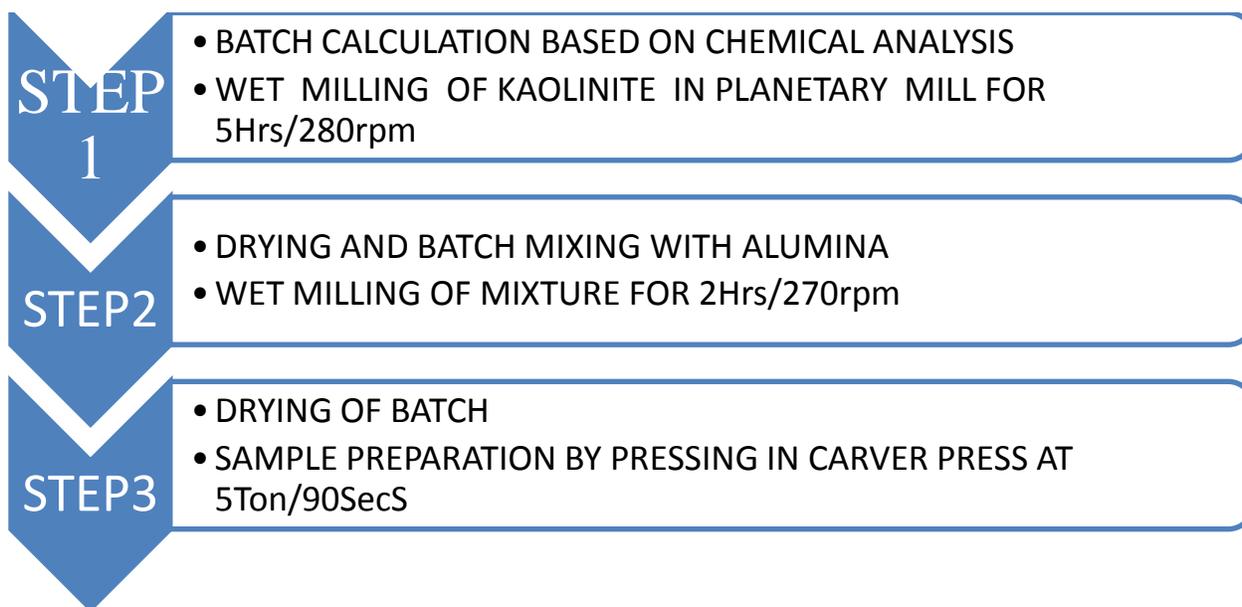
Raw kaolinite + calcined alumina [S1]

Calcined kaolinite (600⁰C) + calcined alumina [S4]

Calcined kaolinite (1000⁰C) + calcined alumina [S2]

Calcined kaolinite (1400⁰C) + calcined alumina [S3]

STEPS FOR BATCH PREPARATION



Initially the only kaolinites for the different batches are taken. Individually they were wet-milled in a planetary ball-mill with Si₃N₄ vessel and balls. They were wet-milled for 5hrs with 280rpm, with 1hr milling and 40mins pause. Then they were dried in a petridis in an oven for 24hrs and after that they were made finer by means of agate mortar.

After that they were mixed with required amount of alumina and again wet milled via the similar way for 2hrs/270rpm. Again the same procedure was followed as previous to obtain a mixture of kaolinite and alumina. The same procedure was carried out for all the four batches.

Each batch was then considered for making specimens for further characterizations. Bars and pallets were prepared. For this individually each batch was mixed with required amount of 4% PVA solution and dried under the lamp to eliminate the probability of any defects in the green body. Finally the batches were considered for pressing. They were pressed in the carver press in 5ton pressure, 45% pump speed with 90seconds soaking time. Pressing of samples was followed by drying for one day and dimensions were measured. And after that they were fired simultaneously in a furnace at 1600⁰C with 2hrs soaking time and steps at 550⁰C/1hr and 980⁰C/1hr. Finally their dimensions were measured using a slide caliper and samples were made ready for further characterizations.

Apparent porosity, bulk density, volumetric shrinkage, flexural strength and XRD analysis were carried out for properties analysis. Apparent porosity refers to the amount of void/pores within a volume of sediment or porous solid. The value of porosity is measured as a percentage.

Apparent porosity & bulk density were measured by water boiling method using Archimedes principle.

CHAPTER 4

RESULTS AND

DISCUSSION

4.1 TG/DSC ANALYSIS OF RAW KAOLINITE

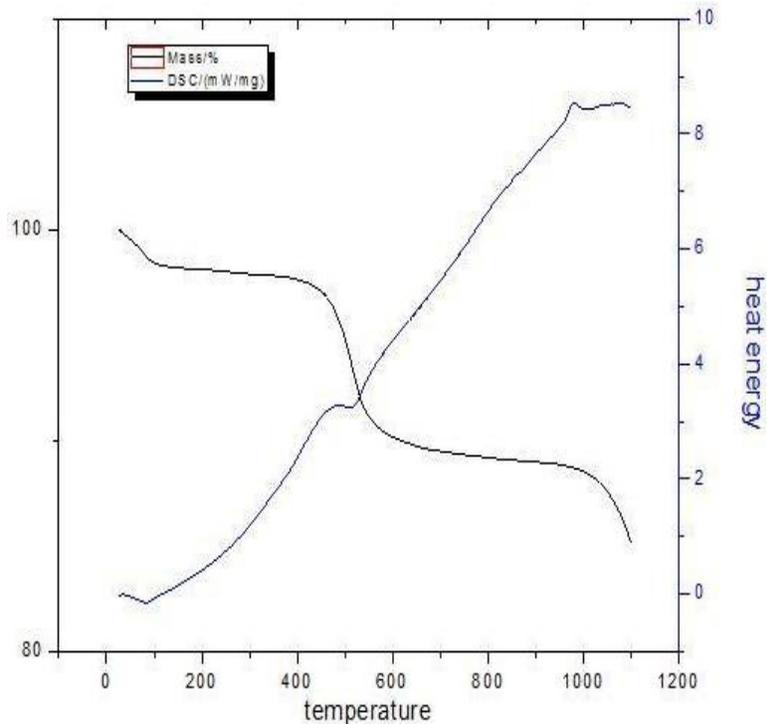


FIGURE-4.1

From the DSC curve i.e. figure 4.1, it can be observed that there are two endothermic and one exothermic peak in the above plot. The first endothermic peak occurs at around 100⁰C due to removal of physically absorbed water. The second endothermic peak is obtained at the temperature near 550⁰C and at this temperature removal of structurally absorbed water occurs, as a result of which meta-kaolin starts forming. Meta-kaolin is dehydrated kaolin and is a meta stable phase. The exothermic peak is found to be at the temperature nearly 980-1000⁰C. This basically happens due to conversion of meta-kaolin into a spinel phase ($2\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$), which is finally converted into mullite.

From the TG plot it can be observed that during the temperature range 450-600⁰C, there is a mass loss and it happens due to the removal of structurally absorbed water. It is an endothermic reaction.

4.2 XRD ANALYSIS OF RAW MATERIALS

XRD analysis of calcined kaolinite (600⁰C, 1400⁰C) and calcined alumina were carried out.

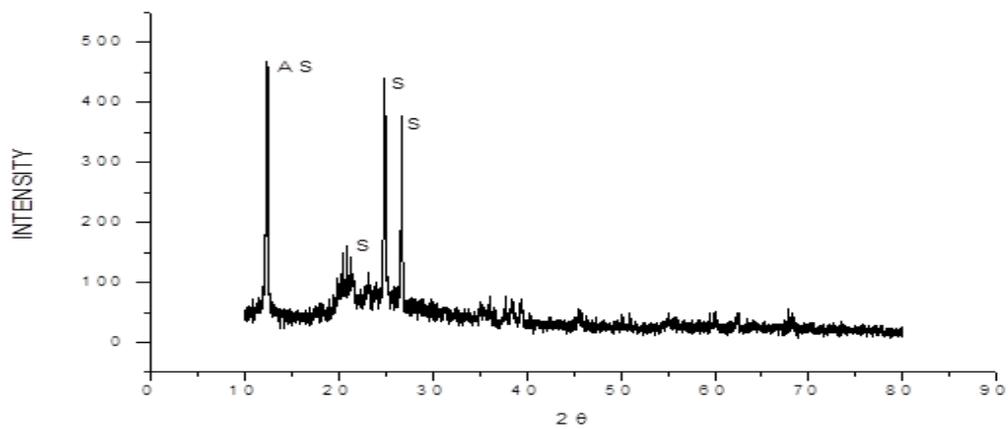


FIG-4.2 (CALCINED KAOLINITE AT 600⁰C)

From the figure 4.2 i.e. XRD plot of calcined kaolinite (600⁰C) it has been observed that as expected the major phases present in are alumino-silicate phases and silica phases.

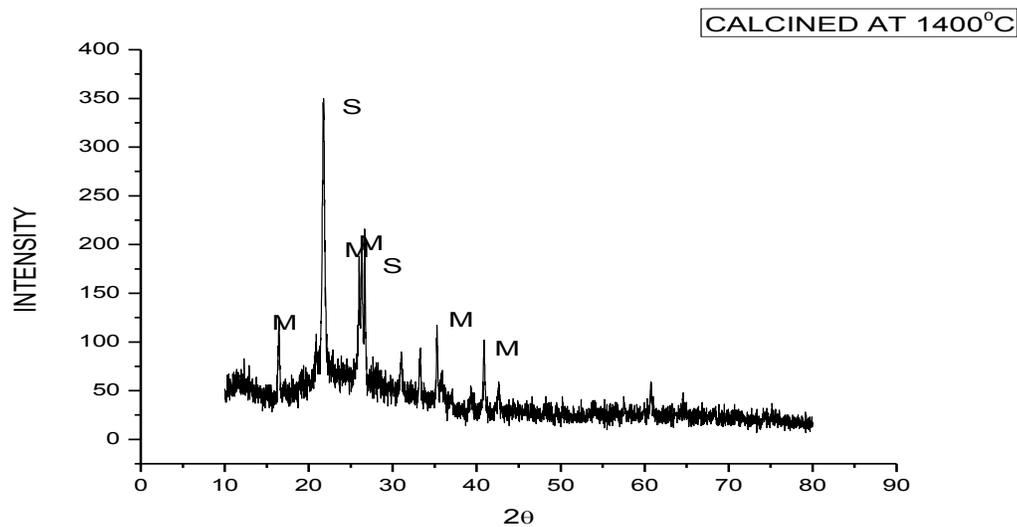


FIG-4.3 (CALCINED KAOLINITE AT 1400°C)

From the figure 4.3 i.e. XRD plot of calcined kaolinite that was calcined at 1400°C it can be seen that here the major phase present is mullite and some amount of crystalline silica is also found

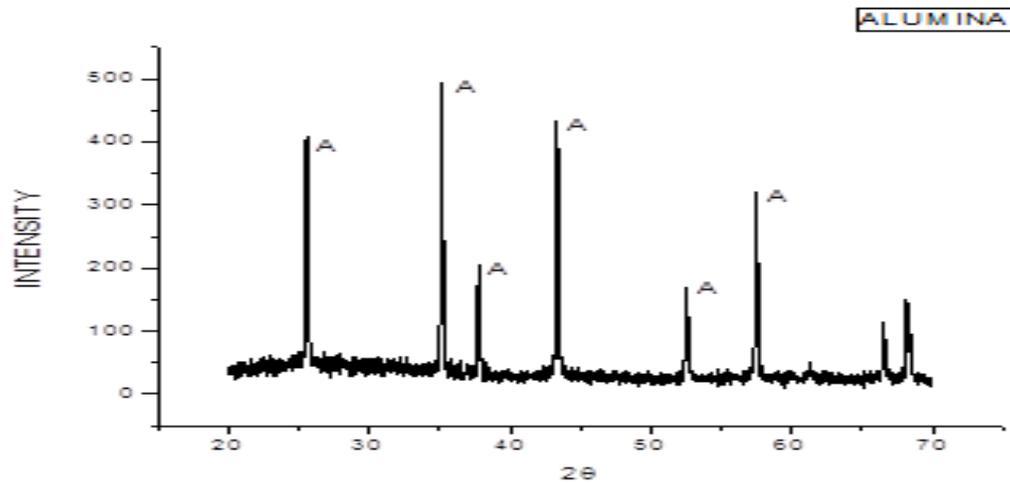


FIGURE-4.4 (CALCINED ALUMINA)

Figure-4.4 is the XRD plot of alumina that was used along with kaolinite for mullitisation. It showed that all the peaks corresponding to pure alumina phase.

4.3 PHYSICAL AND MECHANICAL PROPERTIES

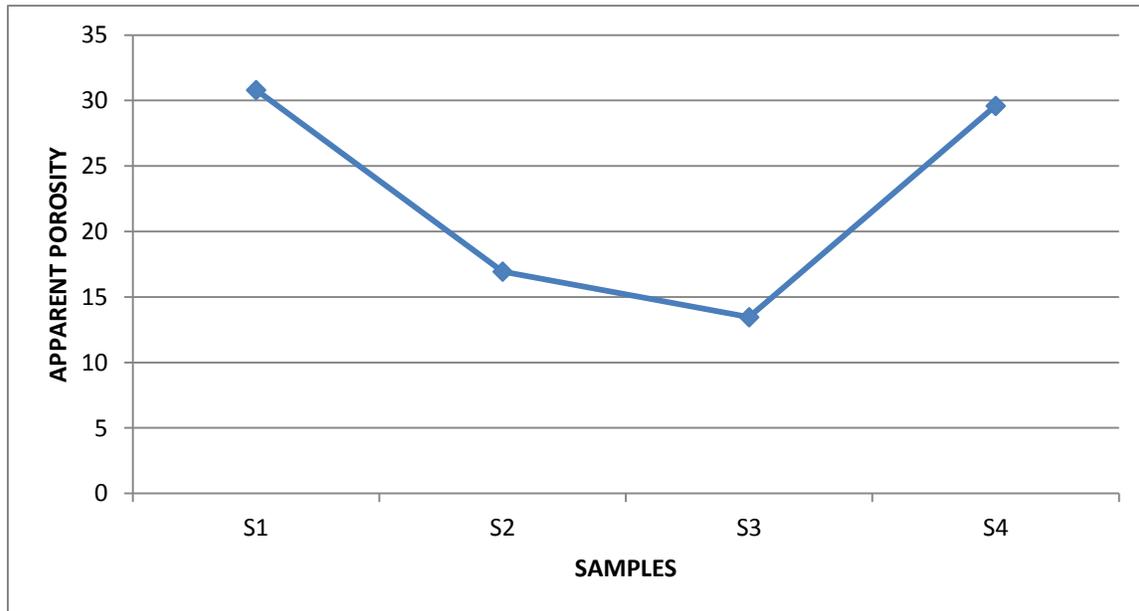


FIGURE-4.5

From fig-4.5 which shows the trend of apparent porosity with variation of calcination temperature. It can be seen that with increase in calcination temperature, when calcined kaolinite is mixed with substantial amount of alumina and fired at 1600°C , it shows lower porosity, on the other hand when the uncalcined kaolinite is mixed with alumina, it shows the highest porosity. In this experimental work minimum porosity is obtained for the sample S3 i.e. the mixture of calcined kaolinite (1400°C) and alumina.

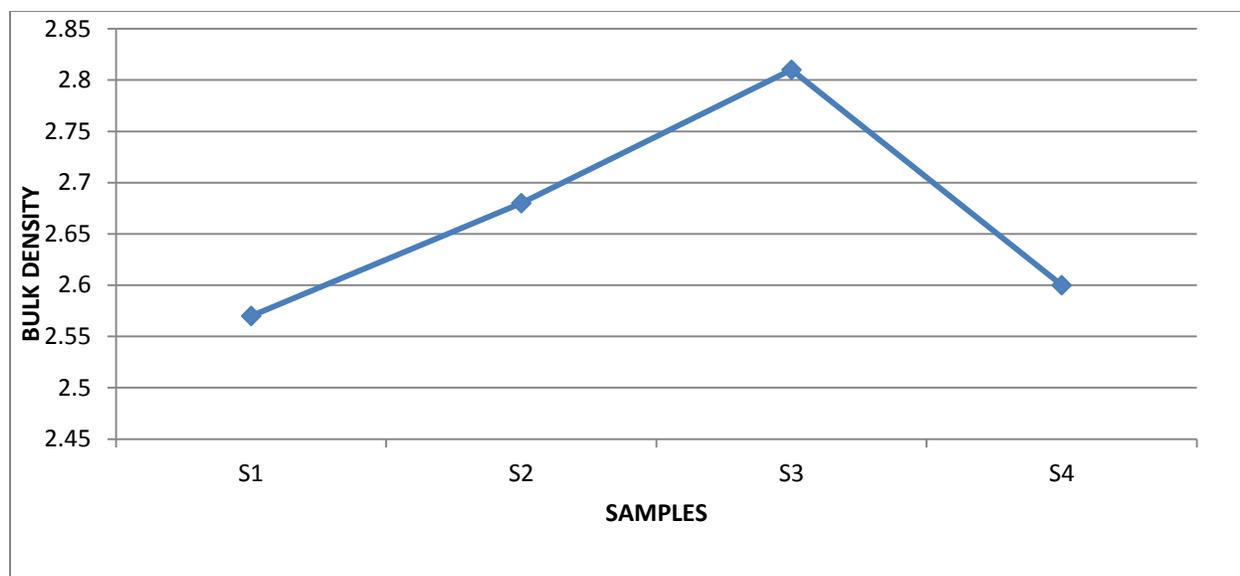


FIGURE-4.6

The figure-4.6 shows the variation of bulk density with calcination temperature of kaolinite in kaolinite and alumina mixture, it is in accordance with apparent porosity data. Here maximum bulk density is achieved for the sample S3 i.e. around 89% of theoretical density achievement, and it is minimum in case of sample S1, in which the raw/uncalcined kaolinite was used. It can be easily observed that, with increase in calcination temperature, the bulk density of the final products increase.

Bi-axial flexural strength of all the samples was measured. It was calculated by using the formula, Flexural strength = $(2F/\pi*D*t)$, here F is the applied force, D is the diameter of the sample and “t” is the thickness.

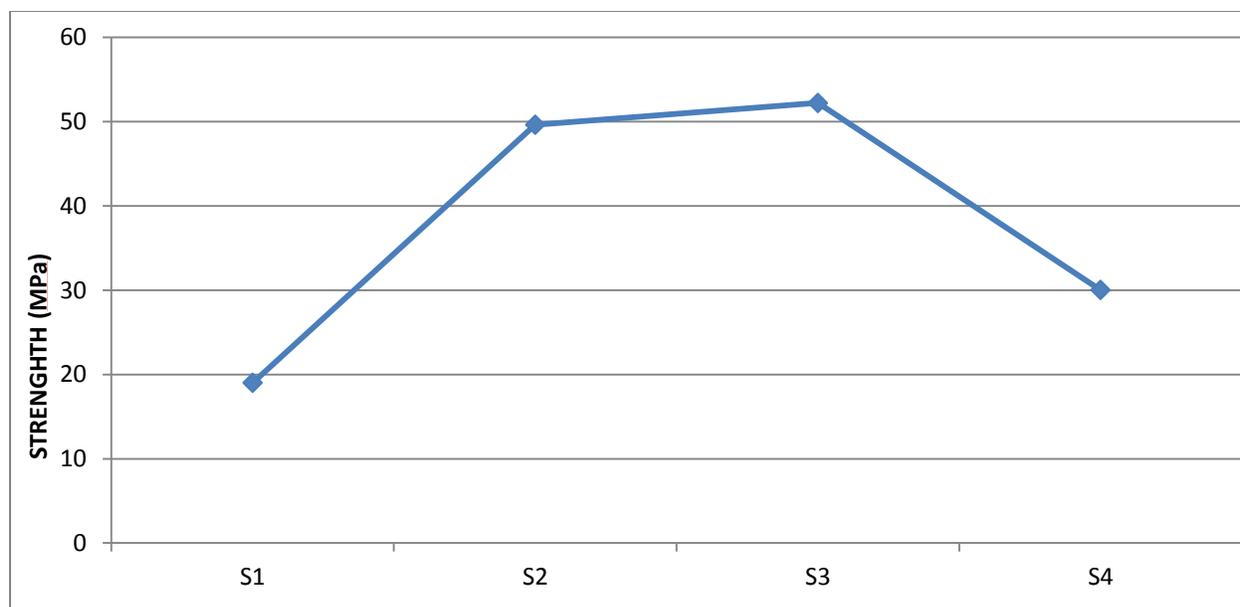


FIGURE-4.7

Figure-4.7 shows the variation of biaxial-flexural strength with calcination temperature. Here it is observed that as expected from the apparent porosity and bulk density data, the sample S3 shows maximum strength and sample S1 shows the minimum. With increase in calcination temperature the strength of materials increase.

	S1	S2	S3	S4
AVEREGE - AP	30.38 %	16.94 %	13.46 %	29.5 %
AVERAGE - BD	2.57	2.68	2.81	2.6
BIAXIAL FLEXURAL STRENGTH	19.02Mpa	49.64Mpa	52.22Mpa	30.04Mpa

TABLE-4.1

So, from the above mentioned data i.e. table-4.1, it can be concluded that, if calcined kaolinite is used instead of raw kaolinite then the density achievement is better and porosity is less, and the plot shows that, the density achievement and reduction in porosity with increase with increase in calcination temperature of kaolinite. It can also be seen from biaxial flexural strength data that, the strength of sample S3 (Higher calcination temperature) is higher and from the pattern we can observe that as the calcination temperature of kaolinite goes up, the strength of mullite is also increased. This is due the fact, when kaolinite is converted into meta-kaolin at 600⁰C, this transformation is actually very slow and that meta-kaolin has a very defect structure I which around 20% space consists of lattice vacancies, produced by temperature induced water release. This may be the reason for lower physical and mechanical properties of kaolinite and alumina mixture.

4.4 XRD ANALYSIS OF FINAL PRODUCTS

In order to find the phases produced in different batches and to study the effect of calcination temperature XRD analysis of the all the final products were carried out.

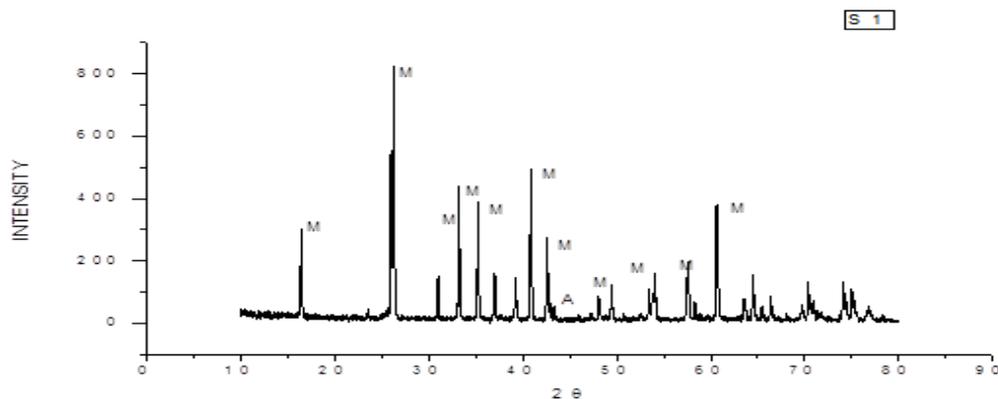


FIGURE-4.8

Figure 4.8 shows the XRD plot of sample S1; here the major phase found was Mullite along with a minor quantity of unreacted alumina.

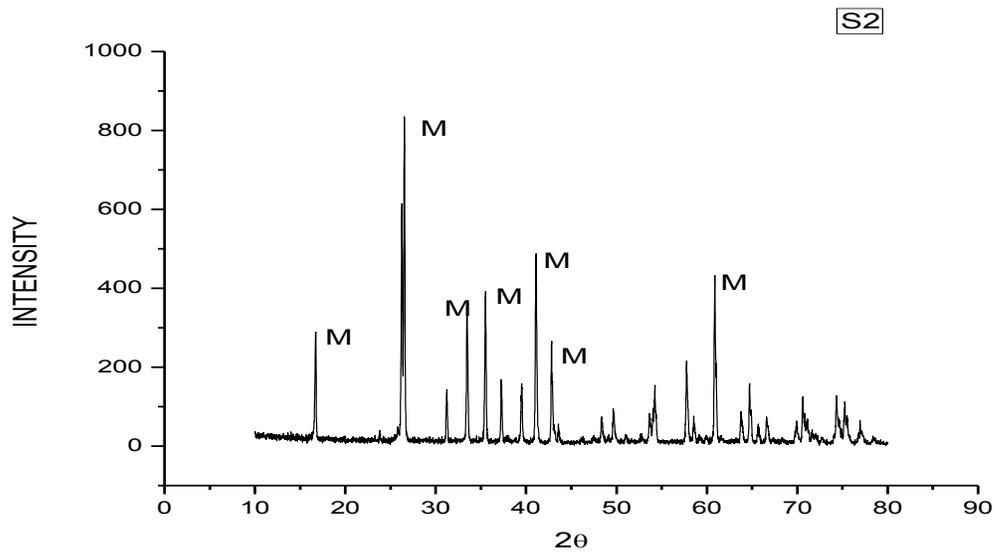


FIGURE-4.9

Figure 4.9 shows the XRD plot of sample S2; here the major phase found was Mullite along with a minor quantity of unreacted alumina. Here almost all the major peaks show the presence of higher amount phases.

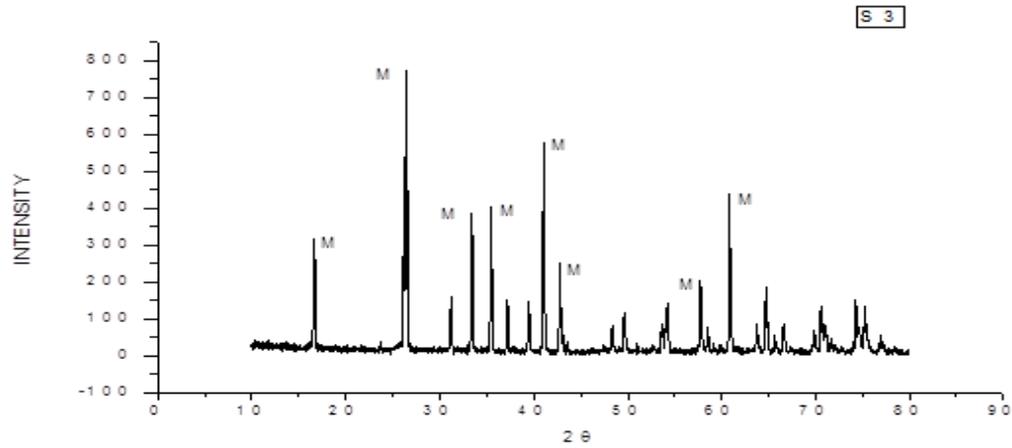


FIGURE-4.10

Figure 4.10 shows the XRD plot of sample S3; here the major phase found was Mullite along with a minor quantity of unreacted alumina. In this sample it has been found that in this sample the amount of mullite phase is highest as compared to others.

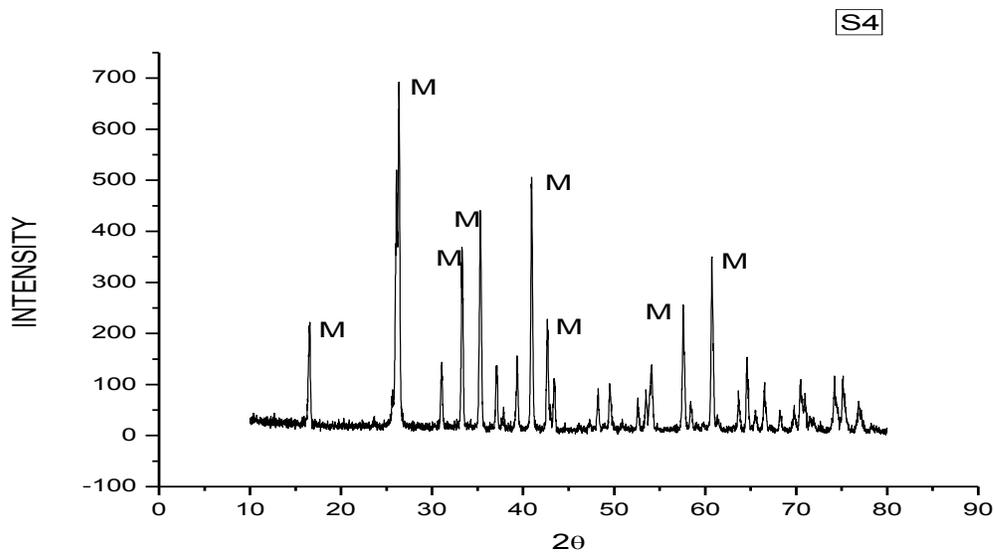


FIGURE-4.11

Figure-4.11 shows the XRD plot of sample S4. It also shows almost the same pattern with other samples.

From the above XRD plots of all the four final products it has been observed that in all the cases mullite has been formed as a major phase and in some cases peaks of alumina of very low intensity have been observed.

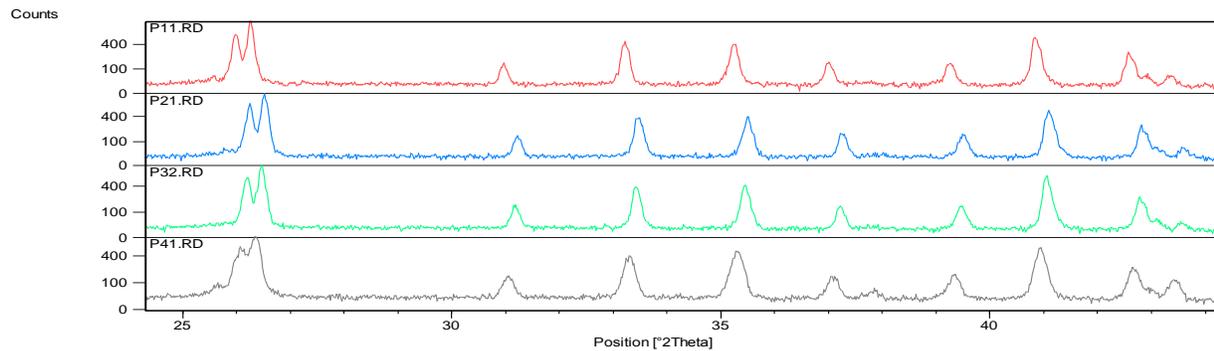


FIGURE-4.12

If all these plots are seen individually then hardly any difference is found in them, all the plots look similar but when analyzed very closely (figure-4.12) it can be seen that there are substantial amount of peak shifting is taking place.

The peak shifting is produced as a result of variation of calcination temperature. The peak shifting takes place towards right as the calcination temperature is increased.

4.5 AMOUNT OF MULLITE PHASE IN DIFFERENT BATCHES

SAMPLES	AMOUNT OF MULLITE
S1	97.64 %
S2	97.66 %
S3	98.84 %
S4	96.94 %

TABLE-4.2

The above quantitative analysis of mullite phase calculation was carried out by peak area calculation analysis. From the above analysis it can be seen that there is hardly any significant difference between the amounts of mullite but the sample S3 has a relatively higher amount of mullite content.

CHAPTER 5

CONCLUSION

CONCLUSION:

- From the experimental data obtained, we can conclude that firing properties of the final sintered body are better when kaolinite is calcined above 600C.
- From the XRD analysis it was found that there is no cristobalite peak and a few peaks of corundum of low intensity in the final sintered sample, that implies the reaction of clay and alumina at 1600C for 2hrs is almost sufficient for conversion into mullite.
- From the XRD analysis it has been found that peak shifting is there. That means crystallite size increases reflecting crystallite growth.
- Compared to the raw kaolinite if calcined kaolinite is used then the density achievement is better and porosity is less, this is due to the fact, when kaolinite is converted into meta-kaolin at 600C, then the transformation is very slow and meta-kaolin has a defect structure, about 20vol% of meta-kaolin consists of lattice vacancies produced by temperature induced water release. This may be the reason for lower physical and mechanical properties of uncalcined kaolinite and alumina mixture.
- From the XRD analysis we can conclude that the amount of mullite formation is almost same in all the cases but relatively a little bit higher in case of 1400C calcined kaolinite. This may be due to the reason that during calcination at 1400C/2hrs it has enough time for complete primary mullitisation and again during firing completely secondary mullitisation occurs, with enough time that increases the mullite content.

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